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(54) Cantilever stylus for use in an atomic force microscope and method of making same.

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Description

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates generally to an atomic force microscope, and more particularly to a flat-cantilever stylus for use in the atomic force microscope and a method of making the stylus. A tip of one of two principal surfaces of the cantilever is used as the stylus.

Description of the Prior Art

Conventionally, a scanning tunneling microscope (hereinafter referred to as STM) has been developed as an instrument capable of observing the surface of a solid body on the order of atoms. However, since the STM detects a tunnel current between a sample and a stylus to observe the sample surface, it is impossible for the STM to observe the surface of an insulator. In order to solve this problem, an atomic force microscope (hereinafter referred to as AFM) for observing the sample surface by detecting forces acting between the sample and the stylus is proposed. Similar to the STM, the resolution of the AFM depends greatly upon the radius of curvature of the tip of the stylus. The less the radius of curvature is, the higher is the resolution. In order to detect minute forces, the AFM requires a cantilever 25 having a stylus 26 formed on the tip thereof, as shown in Fig. 1. In some conventional AFMs, the tip of the cantilever is used as the stylus. A cantilever with an integrated pyramidal tip can be formed by using an etch pit as a mold. The stylus may be made by anisotropic etching. In recent years, styli having a radius of curvature of approximately 300Å at the tip thereof have been obtained. Such a cantilever is known from an article entitled "Improved Atomic force microscope images using microcantilevers with sharp tips" in Applied Physics Letters 57(3), 16 July 1990, pages 316-318.

Although the AFM styli are made by various methods as described above, each method has a problem or problems. When the tip of the cantilever is utilized as the stylus, there are no problems in connection with adhesive properties between the cantilever and the stylus, and the manufacturing processes are comparatively simple. However, the radius of curvature is not less than several thousand angstroms in the ordinary photoetching technique because the radius of curvature of the tip fully depends upon the accuracy of photolithography. Therefore, a microscope manufactured by this method is low in resolution. A maskless etching technique such as FIB (Focused Ion Beam) is required to make the radius of curvature smaller than the aforementioned size, but the manufacturing processes are complicated and the problem of cost occurs. In a stylus made with an etch pit of crystal employed as a mold, although the radius of curvature can be reduced to a comparatively small value, the manufacturing processes are complicated. Furthermore, since the adhesive properties between the stylus and the cantilever are poor, an observation in vibration mode is difficult.

The manufacturing method utilizing anisotropic etching is susceptible to various parameters during etching and is poor in reproducing a stylus configuration.

SUMMARY OF THE INVENTION

The present invention has been developed to overcome the above-described disadvantages.

It is accordingly an object of the present invention to provide an improved cantilever stylus for use in an atomic force microscope, which stylus has a tip having an extremely small radius of curvature.

Another object of the present invention is to provide a method of making the above-described stylus.

In accomplishing these and other objects, the cantilever stylus according to the present invention comprises a cantilever having a fixed end and a free end and having two principal surfaces, and first and second tip portions lying in and defined by the respective principal surfaces at said free end, respectively. The first tip portion has a radius of curvature less than 0.1 µm and protrudes beyond the second tip portion in a direction along its respective said principal surface away from said fixed end so that the first tip portion may be used to observe the sample surface.

A method of making a cantilever stylus according another aspect of the present invention comprises the steps of:

forming on a surface of a substrate a film consisting of a stylus material different from a material of the substrate;

forming on a surface of the stylus material a resist thin film consisting of a material different from the stylus material and having a tip of a given radius of curvature;

etching the stylus material by making use of an isotropic etching technique so that the depth of etching is greater than the radius of curvature of the tip of the resist thin film;

forming the stylus material so that a tip of one principal surface thereof has a radius of curvature less than 0.1µm and protrudes beyond a tip of the opposite principal surface thereof; and

removing the resist thin film and the substrate

adhering to the tips of the stylus material.

Since the tip of the resist thin film is formed by a conventional fine processing technique, the radius of curvature is greater than 0.1μm. The thin film of the stylus material adjacent to the resist thin film is etched from both sides of the tip of the resist thin film using an isotropic etching technique. As a result, when the depth of etching is made greater than the radius of curvature of the tip of the resist thin film, at least the tip of one principal surface of the cantilever adhering to the resist thin film becomes very small in its radius of curvature. Further etching can make the radius of curvature of the tip of the other principal surface very small. Accordingly, the stylus having a radius of curvature less than 0.1μm can be obtained by the conventional photoetching using the tip of one of the principal surfaces as the stylus, without using a fine processing technique on the order of submicrons such as the FIB. This stylus can contribute to an atomic force microscope having a high resolution. In addition, since the tip of the cantilever is used as the stylus, the cantilever and the stylus are formed into a one-piece construction, thereby overcoming the problem of the adhesive properties between the stylus and the cantilever.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other objects and features of the present invention will become more apparent from the following description taken in conjunction with the preferred embodiment thereof with reference to the accompanying drawings, throughout which like parts are designated by like reference numerals, and wherein:

Fig. 1 is a perspective view of a conventional cantilever stylus for use in an atomic force microscope;

Fig. 2 is a perspective view of a cantilever stylus for use in an atomic force microscope according to a first embodiment of the present invention;

Fig. 3 is a side elevational view of the cantilever stylus of Fig. 2 and a sample to be observed;

Figs. 4a to 4c are process diagrams indicative of processes for making the cantilever stylus of Fig. 2;

Fig. 5 is a perspective view of a cantilever stylus according to a second embodiment of the present invention;

Figs. 6a to 6c are process diagrams indicative of processes for making the cantilever stylus of Fig. 5; and

Fig. 7 is a view similar to Fig. 5, showing a modification thereof.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring now to the drawings, there is shown in Fig. 2 a flat cantilever 2 having a stylus 1 integrally formed therewith at the tip thereof according to a first embodiment of the present invention. This flat cantilever 2 is used in an atomic force microscope.

As shown in Fig. 2, the cantilever 2 has one end fixedly connected to a base material 3 and the other free end 4. The cantilever 2 further has two flat principal surfaces. The tip of one principal surface protruding beyond the other is used as the stylus 1.

Fig. 3 shows a relationship between the cantilever stylus 1 and a sample 6 during an observation. In Fig. 3, the cantilever 2 forms an angle of approximately 20° with the sample 6 to be observed. When the cantilever stylus 1 is located in close proximity to a sample surface 7, forces are generated between atoms of the sample surface 7 and those of the cantilever stylus 1, thereby slightly bending the cantilever 2. These forces include attractive van der Waals forces, magnetic forces, electrostatic forces, and repulsive forces. Detecting the bending of the cantilever 2 by any suitable method makes it possible to observe the sample surface 7.

Figs. 4a to 4c depict processes for making the cantilever stylus for use in the atomic force microscope. A SiO_2 film 9 having a thickness of 1 to 2 μm is initially formed on the surface of a Si substrate 8 as a material of the cantilever stylus 1 by thermal oxidation. A WSi_2 film 10 having a thickness of 0.1μm is then formed on the surface of the SiO_2 film 9 as a resist film. The WSi_2 film 10 is processed into a configuration having a tip 11 by the known photoetching technique (Fig. 4a). The radius of curvature of the tip 11 of the WSi_2 film 10 formed is approximately 0.5μm. Then, the substrate 8 is submerged in a buffer etching solution (mixed solution of one volume of HF and six volumes of NH_4F) to perform an isotropic etching of the SiO_2 film 9 for 20 minutes with the WSi_2 film 10 serving as the resist film (Fig. 4b). In this embodiment, a tip having a radius of curvature of approximately 300Å is formed. Then, the substrate is submerged in a dilute mixed solution of hydrofluoric acid and nitric acid to remove the WSi_2 film 10 by etching. Thereafter, the cantilever formed is caused to adhere to a base material 13 and the Si substrate 8 is removed by etching to complete the processes required for making the cantilever 2 and the stylus 1. The tip of the stylus 1 has a radius of curvature of 300Å (Fig. 4c).

The material of the stylus and that of the resist film is not limited to the combination of SiO_2 and

WSi_2, but Si_3N_4 or the like may be used as the material of the stylus and a conventional photoresist material may be used as the material of the resist film.

In this embodiment, although the tip of the protruding principal surface of the cantilever is used as the stylus, a considerably small radius of curvature and a high resolution can be obtained by using the tip of the other principal surface as the stylus. In this case, by the use of monocrystal Si as the Si substrate, the base material of the cantilever can be directly obtained from the Si substrate by the anisotropic etching, thereby simplifying the processes.

Fig. 5 depicts a cantilever 15 and a stylus 14 integrally formed therewith according a second embodiment of the present invention. The stylus surface forms an angle of 55° with the cantilever 15.

Figs. 6a to 6c depicts processes for making the cantilever stylus 14 of Fig. 5. An inclined ⟨111⟩ surface 17 of Si is initially formed on a monocrystal Si substrate 16 by the anisotropic etching (Fig. 6a). The ⟨111⟩ surface formed makes an angle of 55° with an ⟨100⟩ surface 18. At this time, the angle of the inclined surface may be controlled by another method. For example, a steep inclination can be obtained by spatter etching, and the inclination can be easily changed through wet-type processes because the amount of side etching changes according to the etching speed. After a SiO_2 film 19 having a thickness of 1 to 2µm is formed on the surface of the substrate 16 by thermal oxidation as the material of the cantilever stylus, a WSi_2 film 20 having a thickness of 0.1µm is formed on the surface of the SiO_2 film 19. The WSi_2 film 20 is processed so that at least the tip 21 is located on the inclined surface by the normal photoetching (Fig. 6b). Thus, a stylus 23 having a radius of curvature of 300Å and making an angle of 55° with a cantilever 22 is formed by the processes as employed in the first embodiment (Fig. 6c). When the inclination of the stylus makes an angle of 45 to 90° with the cantilever, the tip of one principal surface protruding beyond the other principal surface can be used as the stylus. On the other hand, when the inclination of the stylus makes an angle of 0 to 45° with the cantilever, the tip of the principal surface of the latter can be used as the stylus.

As the material of the stylus and that of the resist film, the combination of the materials as described previously can be used.

In this embodiment, although the cantilever is made by utilizing the lower ⟨100⟩ surface of the Si substrate, the cantilever may be made by utilizing the upper ⟨100⟩ surface of the Si substrate. In this case, as shown in Fig. 7, the tip 24 of one principal surface protruding beyond the other principal surface can be used as the stylus at an inclination in the range of 0 to 90°.

According to the present invention, without using a fine processing technique on the order of sub-microns such as the FIB, a stylus having a radius of curvature of the tip less than 0.1µm and superior adhesive properties to the cantilever can be formed by the conventional photoetching technique. As a result, an atomic force microscope being high in reliability and in resolution and capable of observing a sample surface on the order of atoms can be obtained using the stylus according to the present invention.

Although the present invention has been fully described by way of examples with reference to the accompanying drawings, it is to be noted here that various changes and modifications will be apparent to those skilled in the art. Therefore, unless such changes and modifications otherwise depart from the scope of the present invention, they should be construed as being included therein.

Claims

1. A cantilever stylus (1, 14, 23) for use in an atomic force microscope for observing a sample surface (7) by making use of atomic forces acting between the cantilever stylus (1, 14, 23) and the sample surface, said cantilever stylus (1, 14, 23) comprising: a cantilever (2, 15, 22) having a fixed end, a free end and two opposite principal surfaces; and
first and second tip portions lying in and defined by the respective said principal surfaces at said free end of said cantilever (2, 15, 22), wherein said first tip portion has a radius of curvature less than 0.1 µm and protrudes beyond said second tip portion in a direction along its respective said principal surface away from said fixed end.

2. The cantilever stylus of claim 1, wherein said principal surfaces are parallel and planar and separated by the thickness of said cantilever (2, 15, 22), and wherein said first and second tip portions are separated by the thickness of said cantilever (2, 15, 22).

3. The cantilever stylus of claim 1, wherein said cantilever (15, 22) is bent at an angle of 90° or less at a location between said fixed end and said free end.

4. The cantilever stylus of claim 1, wherein said cantilever (2, 15, 22) and said tip portions are unitary and one-piece and made of SiO_2 and said first tip portion has a radius of curvature of approximately 300 Å.

5. The cantilever stylus of claim 1, wherein said cantilever (2, 15, 22) and said tip portions are unitary and one-piece and made of Si_3N_4.

6. A method of making a cantilever stylus (1, 14, 23) for use in an atomic force microscope, said method comprising the steps of: forming on the surface of a substrate (8, 16) a film (9, 19) consisting of a stylus material different from a material of said substrate (8, 16);
forming on the surface of said stylus material a resist thin film (10, 20) consisting of a material different from said stylus material and having a tip (11, 21) of a given radius of curvature; etching said stylus material by making use of an isotropic etching technique so that the depth of etching is greater than the radius of curvature of said tip (11, 21) of said resist thin film (10, 20); forming said stylus material so that a tip of one principal surface thereof has a radius of curvature less than 0.1 µm and protrudes beyond the tip of the opposite principal surface thereof; and removing said resist thin film (10, 20) and said substrate (8, 16) adhering to said tips of said stylus material.

7. The method of claim 6, wherein an inclined surface (17) is formed at one portion of said substrate (16) so that said inclined surface (17) forms an angle of 0° to 90° with an adjacent portion of said substrate (17), and wherein said tips of said stylus material are formed on said inclined surface (17).

Patentansprüche

1. Freitragende Abtastspitze (1, 14, 23) zur Verwendung in einem Atomkraftmikroskop zum Beobachten einer Probenoberfläche (7) durch Nutzung der zwischen der freitragenden Abtastspitze (1, 14, 23) und der Probenoberfläche wirkenden Atomkräfte, wobei die freitragende Abtastspitze (1, 14, 23) aufweist:
einen freitragenden Teil (2, 15, 22), der ein festes Ende, ein freies Ende und zwei gegenüberliegende Hauptoberflächen aufweist; und
erste und zweite, Spitzen aufweisende Bereiche, die in den jeweiligen Hauptoberflächen liegen und von ihnen am freien Ende des freitragenden Teils (2, 15, 22) definiert werden, wobei der erste Spitzen aufweisende Bereich einen Krümmungsradius von weniger 0,1 µm aufweist und über den zweiten Spitzen aufweisenden Bereich in einer Richtung entlang seiner jeweiligen Hauptoberfläche und weg vom festen Ende hervorspringt.

2. Freitragende Abtastspitze nach Anspruch 1, wobei die Hauptoberflächen parallel und planar sind und durch die Dicke des freitragenden Teils (2, 15, 22) beabstandet sind, und wobei die ersten und zweiten, Spitzen aufweisenden Bereiche durch die Dicke des freitragenden Teils (2, 15, 22) beabstandet sind.

3. Freitragende Abtastspitze nach Anspruch 1, wobei der freitragende Teil (15, 22) an einem Ort zwischen dem festen Ende und dem freien Ende unter einem Winkel von 90 ° oder weniger abgebogen ist.

4. Freitragende Abtastspitze nach Anspruch 1, wobei der freitragende Teil (2, 15, 22) und die Spitzen aufweisenden Bereiche einheitlich und einstückig und aus SiO_2 hergestellt sind, und wobei der erste Spitzen aufweisende Bereich einen Krümmungsradius von etwa 300 Å aufweist.

5. Freitragende Abtastspitze nach Anspruch 1, wobei der freitragende Teil (2, 15, 22) und die Spitzen aufweisenden Bereiche einheitlich und einstückig und aus Si_3N_4 hergestellt sind.

6. Verfahren zum Herstellen einer freitragenden Abtastspitze (1, 14, 23) zur Verwendung in einem Atomkraftmikroskop, wobei das Verfahren die Schritte umfaßt:
Bilden einer Schicht (9, 19) auf der Oberfläche eines Substrats (8, 16), die aus einem Material für die Abtastspitze besteht, welches anders ist als das Material des Substrats (8, 16);
Bilden einer Resist-Dünnschicht (10, 20) auf der Oberfläche des Materials für die Abtastspitze, die aus einem Material besteht, welches anders ist als das Material der Abtastspitze, und die eine Spitze (11, 21) mit gegebenem Krümmungsradius aufweist;
Ätzen des Materials für die Abtastspitze durch Einsatz einer Isotropätztechnik, so daß die Ätztiefe größer ist als der Krümmungsradius der Spitze (11, 21) der Resist-Dünnschicht (10, 20);
Formen des Materials für die Abtastspitze, so daß eine Spitze einer Hauptoberfläche davon einen Krümmungsradius von weniger als 0,1 µm aufweist und über die Spitze deren gegenüberliegender Hauptoberfläche vorspringt; und
Entfernen der Resist-Dünnschicht (10, 20) und des Substrats (8, 16), die an den Spitzen des Materials für die Abtastspitze anhaften.

7. Verfahren nach Anspruch 6, wobei an einem Bereich des Substrats (16) eine geneigte Oberfläche (17) gebildet wird, so daß die geneigte

Oberfläche (17) mit einem angrenzenden Bereich des Substrats (17) einen Winkel von 0 bis 90 • bildet, und wobei die Spitzen des Materials für die Abtastspitze auf dieser geneigten Oberfläche (17) gebildet werden.

Revendications

1. Style (1, 14, 23) en porte-à-faux destiné à être utilisé dans un microscope à force atomique pour l'observation d'une surface (7) d'échantillon par utilisation des forces atomiques agissant entre le style en porte-à-faux (1, 14, 23) et la surface de l'échantillon, le style en porte-à-faux (1, 14, 23) comprenant :

   un organe en porte-à-faux (2, 15, 22) ayant une extrémité fixe, une extrémité libre et deux surfaces principales opposées, et

   une première et une seconde partie de bout disposées dans les surfaces principales respectives et délimitées par ces surfaces à l'extrémité libre de l'organe en porte-à-faux (2, 15, 22), la première partie de bout ayant un rayon de courbure inférieur à 0,1 µm et dépassant de la seconde partie de bout dans la direction de sa surface principale respective du côté opposé à l'extrémité fixe.

2. Style en porte-à-faux selon la revendication 1, dans lequel les surfaces principales sont parallèles et planes, et elles sont séparées par l'épaisseur de l'organe en porte-à-faux (2, 15, 22), et la première et la seconde partie de bout sont séparées par l'épaisseur de l'organe en porte-à-faux (2, 15, 22).

3. Style en porte-à-faux selon la revendication 1, dans lequel l'organe en porte-à-faux (15, 22) est courbé d'un angle de 90• ou moins à un emplacement compris entre l'extrémité fixe et l'extrémité libre.

4. Style en porte-à-faux selon la revendication 1, dans lequel l'organe en porte-à-faux (2, 15, 22) et les parties de bout sont formés en une seule pièce et constitués de SiO_2 et la première partie de bout a un rayon de courbure d'environ 30 nm (300 Å).

5. Style en porte-à-faux selon la revendication 1, dans lequel l'organe en porte-à-faux (2, 15, 22) et les parties de bout sont formés en une seule pièce et constitués de Si_3N_4.

6. Procédé de fabrication d'un style en porte-à-faux (1, 14, 23) destiné à être utilisé dans un microscope à force atomique, le procédé comprenant les étapes suivantes :

   la formation, à la surface d'un substrat (8, 16), d'un film (9, 19) constitué d'une matière de style différente d'une matière du substrat (8, 16),

   la formation à la surface de la matière du style d'un film mince (10, 20) d'une matière de réserve différente de la matière du style et ayant un bout (11, 21) de rayon de courbure déterminé,

   l'attaque de la matière du style par utilisation d'une technique d'attaque isotrope afin que la profondeur d'attaque soit supérieure au rayon de courbure du bout (11, 21) du film mince (10, 20) de la matière de réserve,

   la formation de la matière du style afin qu'un bout d'une surface principale ait un rayon de courbure inférieur à 0,1 µm et dépasse du bout de la surface principale opposée, et

   l'extraction du film mince (10, 20) de la matière de réserve et du substrat (8, 16) adhérant aux bouts de la matière du style.

7. Procédé selon la revendication 6, dans lequel une surface inclinée (17) est formée dans une première partie du substrat (16) afin que la surface inclinée (17) forme un angle de 0 à 90• avec une partie adjacente du substrat (17), et dans lequel les bouts de la matière du style sont formés sur la surface inclinée (17).

Fig. 1 PRIOR ART

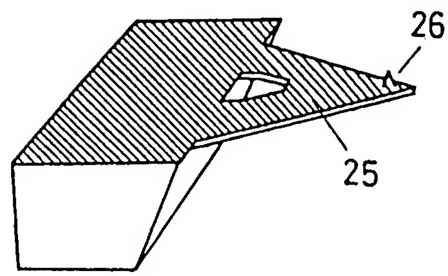

Fig. 2

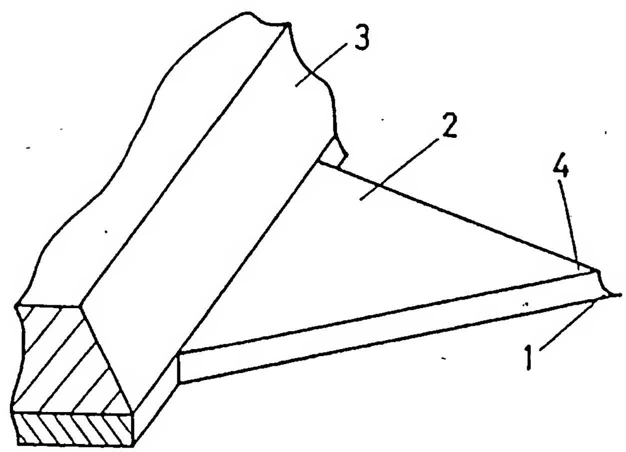

Fig. 3

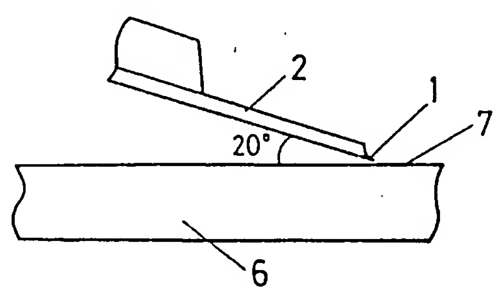

Fig. 4a

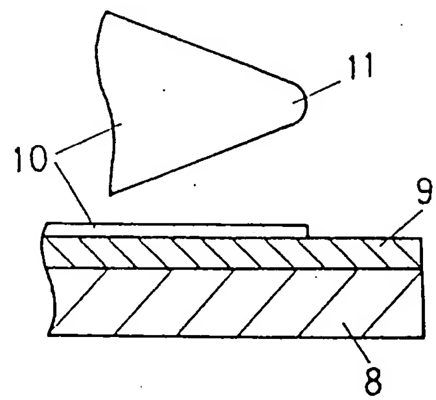

Fig. 4b

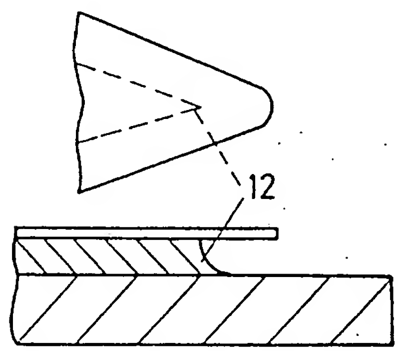

Fig. 4c

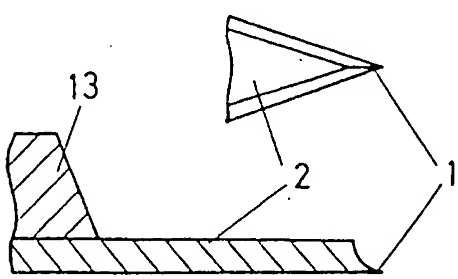

Fig. 5

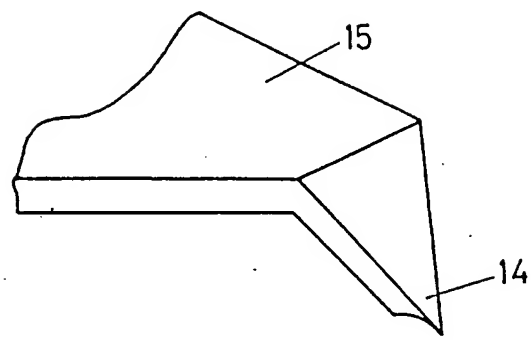

Fig. 7

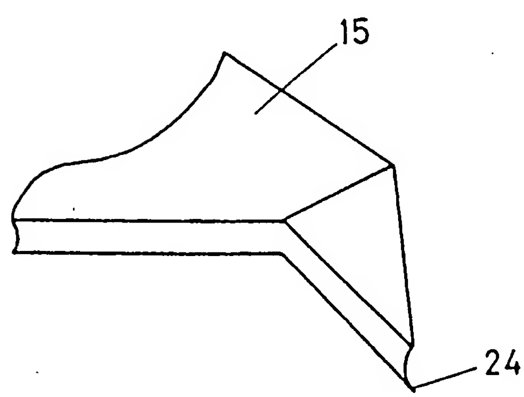

Fig. 6a

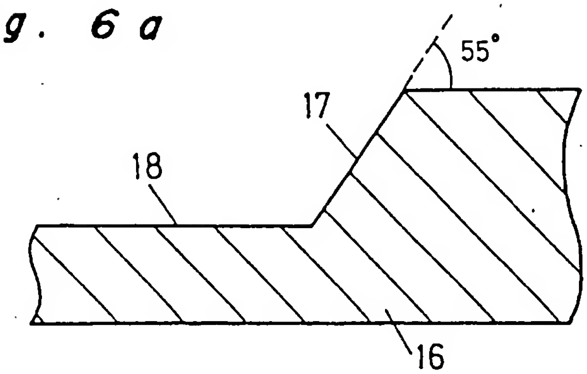

Fig. 6b

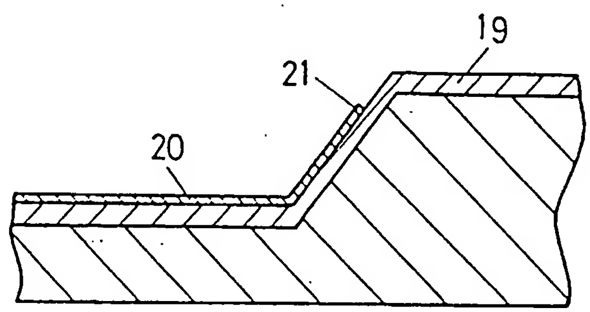

Fig. 6c